Laboratory experiments & tests

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A-Physical Properties:

The Test	***The suitable range***
A1. Color	Colorless
A2. Taste	accept
A3. Odor	Odorless
A4. pH	(6.5 - 8.5)
A5. T.D.S	(≤ 1000)
A6. Turbidity	(≤ 1)
A7. Temperature	(--)
A8. Conductivity	(--)

B- Chemical Experiments:

B1- Free Chlorine:

1-By Using Orthotoluidine: by comparing the yellow color appeared.

2-By Using DPD. Tablets: by comparing the pink color appeared.

The chemicals preparation

-Orthotoluidine:

1gm Orthotoluidine dissolve in 100ml HCl then complete to 500ml with distilled water.

B2-Chlorides: (Cl^-) Max. Range (٢٥٠ ppm)

-Take 50 ml of sample +1 ml potassium chromate (K_2CrO_4) then titrate with silver nitrate ($Ag\ NO_3$ - 0.0141N)

Calculations:

$$Cl^- \text{ (mg/L)} = V_{\text{Titration (AgNO3)}} \times 10$$

The chemicals preparation

-Potassium Chromate (K_2CrO_4):

Dissolve 50 gm in one liter distilled water

-Silver nitrate ($Ag\ NO_3$ - 0.0141N):

Dissolve 2.395 gm in one liter distilled water Or 1.1975 gm in 500 ml distilled water.

B3-Alkalinity: **Max. Range ( )**

- **[Take 50 ml of sample + drops of phenol phethaline then titrate with sulfuric acid (H_2SO_4 – 0.02 N)] color change from pink to colorless the value of titration volume = *V1*.**
- **Take 50 ml of sample + drops of Methyl Orange then titrate with sulfuric acid (H_2SO_4 – 0.02 N) color change from yellow to orange the value of titration volume = *V2***

Calculations

P =V1× 20 (Ppm = mg/L)
T =V2× 20 (ppm = mg/L)

While:

V1 = the value of titration volume by use ph.ph.
V2 = the value of titration volume by use M.O.

- The value of alkalinity is produced as the following table:

Result of titration	Hydroxide alkalinity As $CaCO_3$	Carbonate alkalinity As $CaCO_3$	Bicarbonate alkalinity As $CaCO_3$
P = 0	0	0	T
P < 1/2T	0	2P	(T – 2P)
P = 1/2T	0	2P	0
P > 1/2T	(2P – T)	2(T – P)	0
P = T	T	0	0

The chemicals preparation

-Phenol phethaline: (dissolve 1gm in 1 liter (1: 1 ≡ distilled water: ethanol)).
-Sulfuric acid (0.02 N):

- **Take 27.24 (H_2SO_4 - 98%) then complete by distilled water to 1 liter ≡ (1N H_2SO_4).**
- **Then take 20 ml from (1N H_2SO_4) then complete to 1 liter by distilled water.**

Note: add the acid to water

B4-Hardness: ***Max. Range (500 ppm)***

a- Total Hardness:

Take 50ml of sample +1ml ammonium Buffer + few crystal of *EBT* then titrate with EDTA the color change from pink to blue.

Calculations:

$$\text{Total Hardness (mg/L)} = V_{\text{titrated EDTA}} \times 20$$

b- Calcium Hardness: ***Max. Range ( )***

Take 50ml sample + 1ml Sodium hydroxide + few crystals of Meroxide then titrate with EDTA the color change from yellow to orange.

Calculations:

$$\text{Calcium Hardness (mg/L)} = V_{\text{titrated EDTA}} \times 20$$

c- Magnesium Hardness: ***Max. Range ( )***

$$Mg^{++} \text{ Hardness (mg/L)} = \text{Total Hardness (mg/L)} - Ca^{++} \text{ Hardness (mg/L)}$$

- **Calcium Ion (Ca^{++}):**

$$Ca^{++} \text{ (mg/L)} = Ca^{++} \text{ Hardness (mg/L)} \times 0.4$$

- **Magnesium Ion (Mg^{++}):**

$$Mg^{++} \text{ (mg/L)} = Mg^{++} \text{ Hardness (mg/L)} \times 0.24$$

The chemicals preparation

-Ammonium Buffer:

Dissolve 67.5gm NH_4Cl in 570ml NH_4OH then dilute to 1liter by distilled water.

-EBT:

Mix 0.5gm of the dye in 100gm NaCl.

-Sodium hydroxide (2N - NaOH):

Dissolve 80gm in 1liter distilled water.

-EDTA:

Dissolve 3.723gm in 1 liter distilled water.

B5-Sulfates: (SO_4^{--}) Max. Range (250 ppm)

Take 5ml of acid salt + 5ml of $BaCl_2$ + 100ml of sample let the solution 2 min. in air to stand then measure the turbidity & determine the concentration of (SO_4^{--}) of the sample by using stander relation curve between a known concentrations of sulfate samples & the measurement of there turbidities as shown in the figure (I) below:

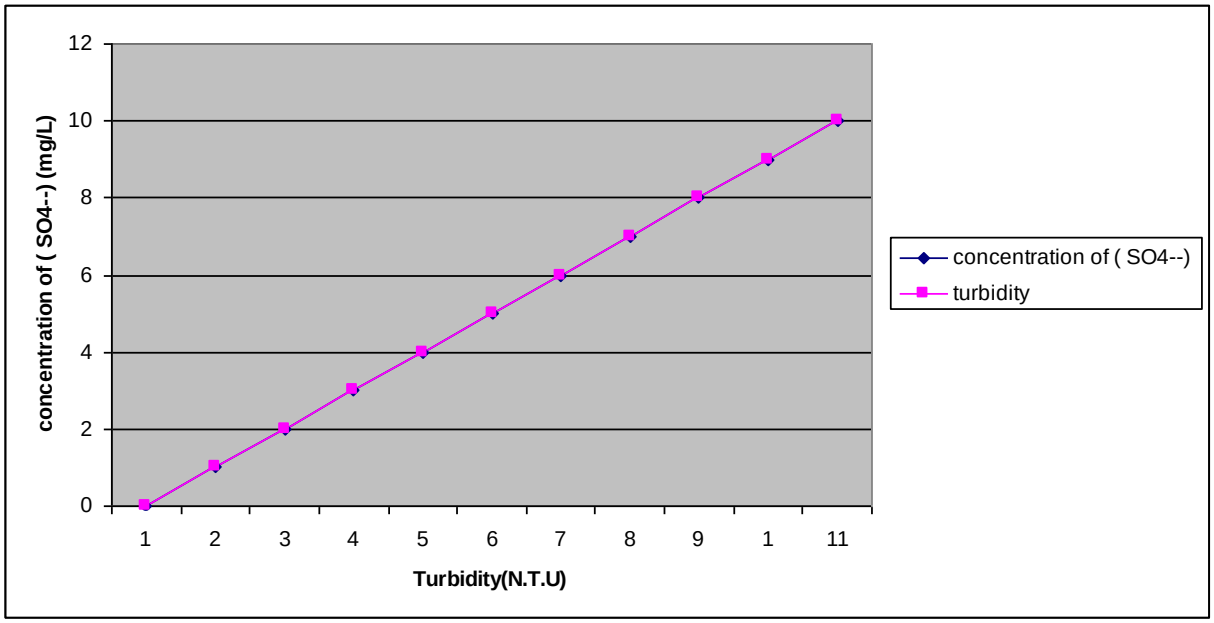

Figure (I)

By making standard deferent concentrations of (SO_4^{--}) salt (known) and measuring the turbidity, then draw the curve above

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The chemicals preparation

- **Acid salt:**

Dissolve 240gm NaCl in 20ml conc. HCl then complete to 1liter by distilled water.

- **$BaCl_2$ (10%):**

Dissolve 100gm in 1 liter distilled water.

[Note: you can use spectrophotometer in stead of turbid meter & measure absorptions of the solutions using wave length (420nm).]

B6- Iron: (Fe^{++}) Max. Range (0.3 ppm)

- **Take 50ml of sample in a (150 or 100ml) beaker + few drops of conc. HCl**
- **Put the solution on the heater & let boiling to evaporate the half of solution.**
- **Then put 1ml potassium permanganate ($KMnO_4$) then complete to 50ml by distilled water then put 5ml ammonium thiocyanate then compare the color by standard solutions of iron with deferent concentrations then determine the concentration of the sample.**

[Note: you can use spectrophotometer & measure absorptions of the solutions using wave length (510nm) by use a curve like figure (1).]

The chemicals preparation

-Ammonium thiocyanate:
Dissolve 20gm in 1 liter distilled water.

- potassium permanganate ($KMnO_4$):
Dissolve 6.32gm 1 liter distilled water.

B7-manganese: (Mn^{++}) Max. Range (0.4 ppm)

- **Take 50ml of sample in (100 or 150ml) beaker +drops of special solution of manganese, then boil & put powder ammonium per sulfate, then pink color appears if there are (Mn^{++}) then compare the color by standard solutions of manganese with deferent concentrations then determine the concentration of the sample.**

[Note: you can use spectrophotometer & measure absorptions of the solutions using wave length (525nm) by use a curve like figure (1).]

The chemicals preparation

-Special solution of manganese:

Dissolve 75gm $HgSO_4$ in [400ml HNO_3 + 200ml distilled water] then add 200ml phosphoric acid 85% + 0.035gm $AgNO_3$ then complete to 1 liter by distilled water.

Note: add the acid to water

B8- Ammonia: (NH_3) Max. Range ( )

Put 1ml Nessler solution then complete to 50ml with the sample, yellow color appears proved that there are ammonia in the sample then compare the color by standard solutions of ammonia with deferent concentrations then determine the concentration of the sample.

[Note: you can use spectrophotometer & measure absorptions of the solutions using wave length (360nm) by use a curve like figure (1).]

The chemicals preparation

-Nessler solution:

Dissolve 100gm HgI_2 &70gm KI in a few distilled water + [160gm NaOH in 500ml distilled water then (*you can warm if you need*) then cool the solution] mix slowly with stirring.

Note:
-Addition must be step by step.
-Store Nessler solution in borosilicate glass ware with rubber stopper & keep it far from sunlight (it's stable for one year).

B9- Nitrate: (NO_3^-) Max. Range ( )

Add 1ml of Diphenyl amine on a tube which contain 50ml sample then add on the wall of the tube drops of conc. (H_2SO_4), "The violet ring which appears on the surface" is proving the present of nitrate.

The chemicals preparation

-Diphenyl amine:

Dissolve 2.5gm in (500ml conc. Sulfuric acid + 100ml water free of ammonia)

B10- Nitrite: (NO_2^-) Max. Range ( )

Take 1ml sulfanilic acid +1ml α-naphthyle amine in a(50ml) tube then complete by sample to 50ml, the red color appear is proving the presence of Nitrite, then compare the color by standard solutions of ammonia with deferent concentrations then determine the concentration of the sample.

The chemicals preparation

-Sulfanilic acid:

Dissolve 8gm of acid pure salt in 750ml free water of ammonia + 250ml glacial acetic acid.

-α naphthyle amine:

Dissolve 5gm in 750ml free water of ammonia + 250ml glacial acetic acid.

[Note: water free of ammonia is prepared by adding 0.1ml conc. H_2SO_4 on (1) liter distilled water & then redistilling].

B11-Dissolved oxygen: (D.O) Max. Range ( )

- Fill 300ml bottle (B.O.D bottle) then add 1ml $MnSO_4$ solution & 1ml alkaline iodide azide reagent.
- When PPT has settled sufficiently add 1ml conc. (H_2SO_4) stopper and mix by inverting several times until dissolution completely.
- Titrate a volume corresponding to 201ml with (0.025M) sodium thiosulfate ($Na_2S_2O_3$) solution by using starch as indicator, (blue color disappears).

Calculations:

For 200ml sample 1ml (0.025M) sodium thiosulfate ($Na_2S_2O_3$) ≡ 1mg (D.O)/L

The chemicals preparation

-manganese sulfate: $MnSO_4 .1H_2O$

Dissolve 360gm in 1 liter distilled water

-Alkaline iodide-azide reagent:

1. **Dissolve 10g in 40ml distilled water with stirrer.**
2. **Dissolve (500mg NaOH + 135g of NaI) or (700g KOH + 150g KI) in distilled water.**
3. **Add (2) to (1) then dilute to 1liter by distilled water.**

-sodium thiosulfate: ($Na_2S_2O_3$) (0.025 M)

Dissolve 6.2g in 1 liter distilled water.

-Starch:

Dissolve 2g + 0.2g salicylic acid in 100ml hot distilled water.

[Note: The bottle mustn't have any air bubble].

B12-biological oxygen demand: (BOD). Max. Range ( )

- **Firstly, prepare dilution water by add 1ml phosphate buffer, 1ml calcium chloride, 1ml magnesium sulfate & 1ml ferric chloride.**
- **Prepare the BOD bottle with a known amount (2, 4, 6, 8… etc) then complete by dilution water.**
- **Measure the dissolved oxygen before incubate make sure you have a blank sample *[blank is a sample with distilled water].***
- **Put the samples in the incubator at 20ºc for 5 days then after the fifth day measure the dissolved oxygen.**

Calculations:

$$BOD5\ (mg/L) = (D1-D2) * (B1-B2)/P$$

As:

- **D1 = D.O of the sample before incubate.**
- **D2 = D.O of the sample after incubate.**
- **B1 = D.O of the blank before incubate.**
- **B2 = D.O of the blank after incubate.**
- **P (dilution coefficient) = volume of BOD bottle / volume of used sample**

The chemicals preparation

-Phosphate buffer:

Dissolve 8.5g of potassium dihydrogen phosphate (KH_2PO_4), 21.75g dipotassium hydrogen phosphate (K_2HPO_4), 33.4g disodium hydrogen phosphate hepta hydrate ($Na_2HPO_4 . 7H_2O$)& 1.7g ammonium chloride (NH_4Cl) in 500ml distilled water then dilute to 1 liter.

-Magnesium sulfate: ($MgSO_4. 7H_2O$)

Dissolve 22.5g in 1 liter distilled water.

-Calcium chloride: ($CaCl_2$)

Dissolve 27.25g in 1 liter distilled water.

-Ferric chloride: ($FeCl_3. 6H_2O$)

Dissolve 0.25g in1 liter distilled water.

-Sodium thiosulfate: ($Na_2S_2O_3$) (0.025 M)

Dissolve 6.2g in 1 liter distilled water.

-Starch solution:

Dissolve 2g + 0.2g salicylic acid in 100ml hot distilled water.

B13- chemical oxygen demand: (COD) Max. Range ( )

1-Mix carefully the contents as in the following table:

Digestion vessel	Sample (ml)	($K_2Cr_2O_4$) (ml)	(H_2SO_4) (ml)	Total final volume
16 × 100 mm	2.5	1.5	3.5	7.5
20 × 150 mm	5	3	7	15
25 × 150 mm	10	6	14	30
Standard 10ml ampoule	2.5	1.5	3.5	7.5

2-Then put in digestion instrument heating at 150°c for 2 hours.
3-Cool to room temperature then add 1-2 drops of ferion indicator.
4-Titrate with standard (0.1M FAS) till color change from blue green. to reddish brown
Do the same steps for a blank sample (distilled water).

Calculations:

$$COD\ (ppm) = [(B-S) \times M_{titration\ of\ FAS} \times 8000] / ml\ of\ sample$$

As:

- **B = the volume of FAS titration of the blank.**
- **S = the volume of FAS titration of the sample.**

The chemicals preparation

-Potassium dichromate: ($K_2Cr_2O_4$) (0.01667M) ***Dry well***

Dissolve 4.903gm in 500 ml distilled water then add 167ml conc. H_2SO_4 & add 33.3 gm $HgSO_4$ mix well & dilute to 1L by distilled water.

-Sulfuric acid reagent: (H_2SO_4)

Add $AgNO_3$ crystals to conc. H_2SO_4 at the rate of: (5.5gm to each 1Kg H_2SO_4).

-Ferion indicator:

Dissolve 1.485 gm 1, 10 phenanthroline monohydrate & 695 mg $FeSO_4.7H_2O$ in distilled water &dilute to 500ml.

-Ferrous Ammonium sulfate (FAS):

Dissolve 39.2 gm $Fe(NH_3)_2(SO_4)_2.6H_2O$ in distilled water, then add 20 ml conc. (H_2SO_4) then cool & dilute to 1L by distilled water.

Another method which determine COD:

-Take 100 ml of the sample then add a known volume of potassium permanganate (*depending on the expected amount of chemicals* and as the table following) and a same volume of sulfuric acid.

The TDS value (ppm)	Volume of permanganate (ml)
300	10
1000	20
5000	30
>10000	≥ 50

-Put it in water path for 30 minutes after boiling.
-Add a same volume of ammonium oxalate.
-Titrate with permanganate until pink color appear

Calculations:

The volume of permanganate ≡ the consumed oxygen

The chemicals preparation

-Ammonium oxalate: (0.028N)
Dissolve 0.888gm in 1L distilled water.
-Potassium permanganate ($KMnO_4$):
Dissolve 0.4938 gm in 1L distilled water.
-Sulfuric acid (H_2SO_4): (1:3)
1volume sulfuric to 3 volume distilled water.